CALIBRATED SCALE IN THE NANOMETER RANGE FOR TECHNICAL DEVICES USED FOR THE HIGH-RESOLUTION OR ULTRAHIGH-RESOLUTION IMAGING OF STRUCTURES

This is a division of U.S. Application No. 08/795,622, filed February 6, 1997 (Attorney Docket No. 02345/17).

Field of the Invention

The present invention relates generally to calibrated scales, and more particularly to scales for use for calibrating very sensitive microscopes, such as electron microscopes.

5

Related Technology

The following articles have to do thematically with the present invention and are based on related art.

10   1.   Technical data sheets from the firms Leitz (Wetzlar) and Zeiss (Oberkochen). The technical data sheets include a description of metal patterns in the micrometer range (0.5 μm to 10 μm), which are lithographically defined on substrates. When objects are measured in the nanometer range, these patterns can be scaled, it then being necessary, however, to take substantial errors into account.

15

2.   Technical data sheets from the firm LOT (Darmstadt). The technical data sheets from the firm LOT describe patterns that are produced lithographically in semiconductor materials. Numbered among these are, for example, line- or cross-lattice structures in silicon, which can be defined with the help of holographic

20   methods. In this case, the lines are spaced apart by 300 nm or 700 nm.

3.   Technical data sheets from the firm Plano (Marburg). The technical data sheets from the firm Plano describe patterns fabricated using electron beam methods and aluminum vapor deposition on silicon substrates. The aluminum tracks produced have a thickness as well as minimal width of 500 nm.

5

The scales described under numbers 2 and 3 must also be scaled before they can be used in the nanometer range. This has its inherent drawbacks.

The following articles are mentioned as examples of analysis methods based on X-ray diffractometry and are hereby incorporated by reference herein:

10

1.   Appl. Phys., 56(1984), p. 1591.

Speriosu, V.S.; Vreeland, T., Jr.:

"X-ray Rocking Curve Analysis of Superlattices";

15   2.   Springer, Proceedings in Physics 13, Les Houches, France.

Quillec, M.:

"Structural Characterization of Superlattices by X-ray Diffraction";

3.   Phys. stat. sol. (a), 105(1988), p. 197.

20   Baumbach; Brühl; Pietsch; Terauchi:

"Characterization of AlGaAs/GaAs-Superlattices and Thin Layers by X-ray Diffraction".

The following sources contain model calculations, which can be used to

25   evaluate the experimental data of the analysis methods, and are hereby incorporated by reference herein:

1.   Acta Cryst., A42(1986), p. 539-545.

      Bartels, W.J.; Hornstra, J.; Lobeek, D.J.:

      "X-Ray Diffraction of Multilayers and Superlattices",

5

2.   J. phys. soc. Japan, 26(1969), no. 5, p. 1239   Tagaki, S.:

      "A Dynamical Theory of Diffraction for a Distorted Crystal";

3.   Journal of Crystal Growth, 44(1978), p. 513-517. Hornstra, J.; Bartels, W.J.:

10   "Determination of the Lattice Constant of Epitaxial Layers of III-V-

      Compounds";

      The model equations are based on a formulation published in 1986 (1) for
calculating the reflectivities of Bragg reflections, which is based on the Tagaki-Taupin

15   formalism (2) of the dynamic theory of X-ray diffraction, and on a treatise on the
lattice constants when working with strained epitaxy layers of III-V semiconductors
(3).

      Epitaxial methods can be used, such as molecular-beam epitaxy, to deposit
semiconductor heterostructures, i.e., layers which vary in composition and thickness,

20   one after another on a semiconductor substrate. Examples of the extensive literature
on this subject are:

1.   Springer Publishers, Berlin 1984, p. 88.

      Springer Series in Solid-State Sciences, vol. 53 Weimann, G., et al.:

25   "Two-dimensional Systems, Heterostructures and Superlattices";

2.   J. of Cryst. Growth, 105(1990), p. 1-29.

      Tsang, W.T.:

      "Progress in Chemical Beam Epitaxy";

30

3

3.   H.C. Freyhardt (Editor), Springer Publishers 1980.

Ploog, K.:

"Growth, Properties and Application";

5   4.   Thin Solid Films, 205(1991), pp. 182-212.

Adomi K., et al.:

"Molecular Beam Epitaxial Growth of GaAs and Other Compound

Semiconductors".

10   These four references are also incorporated by reference herein.

What all the named literature sources on epitaxial methods have in common is

that the individual layer thicknesses are able to be determined from the growth rates

and from various in-situ control methods, such as RHEED and ellipsometry.

However, they do not attain the accuracy achieved by the present invention.

15   Some early pioneer works are cited as examples from the voluminous

literature on characterizing semiconductor heterostructures, mostly on the basis of

photoluminescence, and as a further example of an analysis method:

1.   "Festkörperprobleme" [Advances in Solid State Physics],

20   vol. XV (1975), p. 21., Dingle, R.:

"Confined Carrier Quantum States in Ultrathin Semiconductor

Heterostructures";

2.   Reviews of Modern Physics, 54 (1982), p. 437. Ando, T.; Fowler, A.B.; Stern,

25   F.:

"Electronic properties of two-dimensional systems."

These two references are also incorporated by reference herein.

The experimental data ascertained from the "photoluminescence" analysis

30   method are evaluated, for example, by simulating photoluminescence spectra, as

described by the following article, which is also incorporated by reference herein:

IEEE J. Quantum Electron., 26(1990), p. 2025.

5 Jonsson, B.; Eng, S.T.:

"Solving the Schrödinger Equation in Arbitrary Quantum-Well Potential Profiles Using the Transfer Matrix Method".

Summary of the Invention

10 The present invention is directed to the calibration of a spacial scale (spatial coordinates) of technical devices, which work on the basis of high-resolution and ultrahigh-resolution imaging processes. These are imaging processes based on particle flows, such as scanning electron microscopy, scanning transmission electron microscopy, or scanning probe microscopy (atomic force microscopy, scanning

15 tunneling microscopy).

The technical task at hand is to provide a scale which will enable the technical devices mentioned above to be calibrated with very high precision. The present invention achieves this objective by enabling scales to be manufactured and calibrated in the nanometer range.

20 More particularly, the present invention provides a scale in the nanometer range for technical devices which are used for the high-resolution or ultrahigh-resolution imaging of structures characterized in that:

-a to construct the scale, at least two different crystalline or amorphous materials

25 are used for the heterolayer structure, which are easily distinguished from one another by their contrast when they are imaged using high-resolution or ultrahigh-resolution imaging methods; that

-b the different crystalline or amorphous material layers used are deposited by means of a material deposition method in the deposition direction, one after

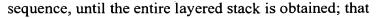30 another in alternating sequence onto a substrate material, as a heterolayer

5

sequence, until the entire layered stack is obtained; that

-c   the heterolayer sequence is characterized experimentally using an analysis method that is sensitive to the layer thicknesses of the heterolayer sequence, the applied analysis method being independent of the high-resolution or ultrahigh-resolution imaging methods used in the technical devices for which the scale is manufactured; that

-d   the experimental data obtained from the analysis method are evaluated and recorded, to make it possible to define the spacings between equivalent hetero-interfaces; and that

-e   subsequently, right after the step according to point b or the step according to point c, the heterolayer structure of the various materials is exposed by splitting open the heterolayer sequence in the deposition direction.

Brief Description of the Drawings

Fig. 1 shows a scale constructed from various materials according to the present invention.

Fig. 2 shows a scale constructed from two heterogeneous materials A and B according to the present invention.

Fig. 3 shows an example of a scale made in accordance with the present invention.

Fig. 4 shows experimental data and simulation or model data showing the reflection at various degrees for a scale made in accordance with the present invention. The simulation or model data is moved downward from its actual position to show its correspondence with the experimental data.

Fig. 5 shows a flowchart of a method for making a scale according to the present invention

Detailed Description

The present invention is directed to manufacturing and calibrating a scale in the nanometer range. To construct the scale, at least two different crystalline or

6

amorphous materials (e.g., material A, B, and C, etc.) are used in accordance with the present invention, as shown in Figure 1 and step 102 of Fig. 5. The materials used differ particularly with respect to their compositions, making them easily distinguishable from one another by their contrast when they are imaged using high-resolution or ultrahigh-resolution imaging methods. In the following, z denotes the deposition direction of the deposition process (vertical direction). The interfaces of the heterolayer structure lie parallel to the xy-plane (lateral directions). The scale 10 has m layers, identified generally as 101, 102, 103, 104, etc., formed on a substrate 12. The layers have thicknesses L.

The different materials are deposited as thin layers on a substrate using a material deposition process (Step 104 of Fig. 5). Preferably, the deposition methods used are those which result in a best possible homogeneity of the heterolayer structure in all spatial directions with respect to composition and lattice mismatch of the individual layers. The heterolayer structure is deposited on the carrier material, for example a substrate, in the deposition direction, the different material layers being deposited one after another in alternating sequence on to the substrate as a heterolayer sequence, until the entire layered stack is obtained. The thickness of the individual layers lies in the nanometer range, preferably less than 25 nanometers. The thickness of the heterolayer sequence lies in the range of approximately 20-200 nanometers. In terms of process engineering, the object of the present invention is able to be realized, for example, by growing a heterolayer structure of thin semiconductor material layers on a substrate using epitaxial methods, such as molecular beam epitaxy, organometallic molecular beam epitaxy, or organometallic gas-phase epitaxy.

Following fabrication, the thus produced heterolayer sequence is characterized experimentally using at least one analysis method. (Step 108 of Fig. 5) The analysis method selected must be sensitive to the individual layer thicknesses of the heterolayer sequence and ensure a high degree of precision. The analysis method(s) must be independent of the high-resolution or ultrahigh-resolution imaging methods used in the technical device(s) to be calibrated. In connection with the X-ray diffractometry analysis method, it is especially expedient to produce a periodic,

7

crystalline multi-layered structure (e.g. multiple quantum film structure) or a multiple layer having a superlattice structure. Important structural dimensions are in the case of the multi-layered structure the period thickness and, in the case of the superlattice structure, the superlattice period.

5   By evaluating the experimental data obtained from the analysis method(s), the period thickness of the heterolayer sequence, for example, or the individual layers of the heterolayer sequence are determined and recorded, so that the spacings between equivalent or non-equivalent interfaces can be defined.

If a periodic heterolayer structure was determined from the materials A and B,
10   then every second hetero-interface is an equivalent hetero-interface, whereas two adjacent hetero-interfaces are non-equivalent.

Before or after evaluating the experimental data obtained from the analysis method(s), the layer structure of the different crystalline or amorphous materials is revealed by separating the produced heterolayer sequence in the deposition direction.
15   (Step 106 of Fig. 5). If a crystalline heterolayer structure is revealed, it can be cleaved, for example, along the crystal planes which show the deposition direction. The cleaving along the crystal planes can take place once the process of depositing the various materials onto the substrate is complete. Other methods for revealing and exposing the heterolayer structure comprise etching and oblique grinding.

20   The heterolayer sequence is able to be experimentally characterized, i.e., the thickness of some or all of the individual layers is able to be determined through physical analysis methods, such as X-ray diffractometry or photoluminescence.

If X-ray diffractometry is used to analyze, e.g., a crystalline, periodic heterolayer structure, then the accuracy that is attainable in sizing the scale to be
25   manufactured rises with the number of layers deposited on the substrate. The accuracy is further increased by using simulation programs with model calculations to additionally simulate the experimental results of the analysis methods (e.g., X-ray diffractometry).

To enhance the material contrast of the individual thin layers to make the
30   layers distinguishable from one another, appropriate methods, such as chemical

8

etching are applied, as needed.

According to the present invention, one attains a calibrated scale in conjunction with a documented size determination of the heterolayer sequence of the scale. On the basis of these two components, it is possible to calibrate technical devices used for the high-resolution or ultrahigh-resolution imaging of structures.

Exemplary embodiments:

The present invention will be elucidated further on the basis of two exemplary embodiments.

In both exemplary embodiments, monocrystalline semiconductor heterostructures are produced with high local precision in all spatial directions using a suitable epitaxial method. The heterolayer stack being used as a scale is comprised, as illustrated in Figure 2, of a periodic sequence of semiconductor layers of the alternating layer sequences ABABAB having the corresponding layer thicknesses L_A and L_B. The scale 10 in Fig. 2 has layers, identified generally as 101, 102, 103, etc., formed on a substrate 12.

Exemplary embodiment 1 (crystalline, periodic semiconductor structure; analysis method: X-ray diffractometry):

In determining the period length ($L_A + L_B$) in accordance with the present invention, five marginal conditions have to be met:

- The epitaxial semiconductor materials A and B must differ substantially in their lattice constants in the direction of growth. This can be achieved through a selective, different strain in layers A and B in relation to the substrate using modern epitaxial methods.

- The monocrystalline, vertical semiconductor heterostructure must be as constant as possible parallel to the direction of growth (xy plane) in the layer thickness ($L_A + L_B$) within the scope of physically unavoidable fluctuations in the atomic single-layer range. This homogeneity can be reached using present-day epitaxial methods.

9

- There should be an adequate number of periods n of the layer thickness (L_A + L_B) in the structure. The number n of the periods thereby determines the accuracy of the scale, which can be achieved in evaluating the experimental results of the analysis method.

5 - The net strain in the heterolayer sequence of n periods of the period length (L_A + L_B) should be as small as possible in order to rule out stress relaxation in the entire layer sequence.

- The material used must be one for which an appropriate method exists for exposing the heterolayer sequence.

10 - The material system selected must produce sufficient contrast in the high-resolution or ultrahigh-resolution imaging method being used.

The semiconductor material system, aluminum gallium indium-arsenide on indium phosphide (AlGaInAs on InP), selected for the present exemplary embodiment

15 and produced using the epitaxial method of molecular-beam epitaxy, satisfies these conditions. Layer sequences of n periods of the period length (L_A and L_B) were produced by subjecting layer A to a pressure (compressive) strain of about 0.3% to 3% compared to the substrate InP, and by subjecting the second layer B to tensile strain of about 0.3% to 3% compared to the substrate.

20 By this means, one obtains, on the one hand, a greatly varying lattice constant in the two materials A and B; on the other hand, the net strain of the epitaxial layer sequence becomes roughly 0 when the following condition is fulfilled: the degree of strain of layer A multiplied by its thickness is equal to the degree of strain of layer B multiplied by its thickness. In the material system in the present example, the degree

25 of strain is mainly produced by varying the concentration levels of In, the contrast mainly by varying the concentration levels of Al in the A and B layers.

To reveal the produced heterolayer sequence $n(L_A + L_B)$, it is split along certain crystal planes, the crystal planes being formed lying, for example, parallel to the direction of growth.

30 Another method for baring the heterolayer sequence consists in grinding the

10

produced heterolayer sequence obliquely at a defined angle. The angle should be selected so as to ensure that the layers of the heterolayer sequence are distinguished from one another as best possible.

In the two variants described, the contrast can be further enhanced, on the one hand, through a slight etching of the cleaved plane using a wet chemical etching solution, which attacks the materials A and B differently and, on the other hand, with the aid of commercially available image-processing software, which also intensify the material contrast in the technical devices used for high-resolution or ultrahigh-resolution imaging of structures.

To uncover the heterolayer structure, it is also possible to etch trenches into the heterolayer stack opposite the deposition direction, it being necessary for the trench depth to be greater than the overall thickness of the heterolayer stack.

Another possibility for revealing the heterolayer structure consists in etching islands into the heterolayer stack. In this case, the material removed opposite the deposition direction must likewise be greater than the overall thickness of the heterolayer stack.

Figure 3 shows a schematic, cross-sectional representation of a semiconductor heterolayer sequence suitable as a calibrated scale 10, having layers 14 (white) of material A and layers 15 of material B (black). The compositions as well as the dimensions of the individual layers are reproduced alongside the drawing. The structure has n = 10 periods of the thickness $L_A + L_B$. The strain in the material with the smaller band gap amounts to $\Delta a/a = +1.2\%$, the strain in the material with the larger band gap amounts to $\Delta a/a = -1.4\%$.

The accuracy of the thickness determination of the occurring period ($L_A + L_B$) depends essentially on the measuring accuracy of the analysis method used in this example, on the X-ray diffractometry, and on the accuracy with which the physical parameters used in the model calculation (simulation) are known. Measured X-ray diffractometry spectra are thereby compared to mathematically simulated spectra (Figure 4). The simulated curve is shifted to the bottom in this case for clarification purposes. The simulated spectra can be thereby adapted so precisely to the measured

spectra that the errors in the mathematical result for the period of the exemplary embodiment in Figure 3 ($L_A + L_B = 13.0$ nm) lie below 0.1 nm. This means a relative error in the thickness determination of < 0.8%.

5   Exemplary embodiment 2 (crystalline, periodic semiconductor structure; analysis methods; X-ray diffractometry and photoluminescence):

With respect to the way in accordance with the present invention of determining the period ($L_A + L_B$), as well as, additionally, the individual thicknesses (L_A and L_B) in accordance with exemplary embodiment 2, two marginal conditions

10   must be met in addition to those specified in exemplary embodiment 1:

-   The materials must have different band gaps.
-   Of the epitaxially produced semiconductor materials, the one with less band-edge energy must exhibit a direct band gap.

15   The semiconductor material system AlGaInAs on InP selected for the present exemplary embodiment also satisfies these conditions when the right composition parameters are chosen. Within the scope of this second exemplary embodiment, two layer sequences are produced with the structure $n(L_A + L_B)$ and $n(L_A + xL_B)$, L_A being assigned to the layer with less band edge energy that is retained in both layer

20   sequences, while L_B and xL_B are assigned to the layers with greater band edge energy, which were intentionally selected to vary in thickness. The parameter x expresses the difference in the thickness, which amounts, for example, to $x = 3/2$. In this case, the spectral emission signal must not be a function of the variation in the layer thickness of L_B for $x \cdot L_B$ (L_B must be selected to be large enough). The two semiconductor layer

25   sequences are evaluated, as described under exemplary embodiment 1, with the aid of X-ray diffractometry. To ensure that L_A has the same thickness in the two different layer sequences, the emission wavelength characteristic of L_A, given a certain material composition, is determined with the aid of photoluminescence. Given an identical emission wavelength in the photoluminescence spectrum and, thus, identical L_A, two

30   equations are able to be formulated and solved, together with the measuring results of

12

the X-ray diffractometry, with two unknowns, L_A and L_B. Apart from that, all particulars given under the first exemplary embodiment apply, inclusive of the error estimation.

Instead of characterizing by photoluminescence, it is likewise possible to analyze by photoreflection, absorption, or transmission, for example.

When calibrating a microscope using the present invention, a greatest possible number of periods n of layer sequences A and B may be used to minimize reading errors.